"SYNTHESIS AND CHARACTERISATION OF POROUS ACTIVATED CARBON FROM A NEW PRECURSOR (KARANJA OIL CAKE)"

A thesis submitted to the

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In partial fulfillment of the requirements of

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CERTIFICATE

This is to certify that the thesis entitled, "Synthesis and characterization of porous activated carbon from a new precursor (Karanja oil cake)", submitted by Amlan Mahamallik, Roll No.-109CH0475, in partial fulfillments for his requirements for the award of Bachelor of Technology Degree in Chemical Engineering at National Institute of Technology, Rourkela is prepared by him under my supervision and guidance.

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Amlan Mahamallik

Roll No : 109CH0475

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NOMENCLATURE

^0C	Degree Celsius
BET	Brunnauer Emett Teller
Cr	Chromium Metal
MOF	Metal Organic Framework
nm	Nano meter
H_3PO_4	Phosphoric Acid
KOH	Potassium Hydroxide
K_2CO_3	Potassium Carbonate
$ZnCl_2$	Zinc Chloride
HCl	Hydrochloric Acid
NaOH	Sodium Hydroxide
SEM	Scanning Electron Microscope
UV	Ultra Violet
ASTM	American Society for Testing and Materials
M	Moisture Content
A	Ash Content
VM	Volatile Matter
FC	Fixed Carbon
P_0	Saturation pressure of the adsorbate
P	Equilibrium pressure of the adsorbate
V_m	Volume of Gas Adsorbed in the monolayer
c	BET Constant

E_1	Heat of adsorption for the first layer
E_L	Heat of adsorption for higher layers
R	Universal Gas Constant
T	Absolute Temperature
N	Avogadro's Number
S	Adsorption cross-section of the gas being adsorbed
V	Molar Volume of the gas being adsorbed
X	Mass Of adsorbent
FTIR	Fourier Transform Infra-red
KBr	Potassium Bromide
Mg/gm	Milligram per gram
TGA	Thermo Gravimetric Analysis

ABSTRACT

The purpose of this project is to develop a porous activated carbon from a suitable precursor. Precursor used for synthesis of activated carbon was Karanja oil cake. Analysis of the raw material (Proximate and ultimate analysis) were carried out to know the physical properties; whether the raw material was suitable for synthesis of activated carbon not. Phosphoric acid was used for the chemical activation of the raw material. Thermo gravimetric analysis was carried out to know the suitable temperature range for carbonization. Carbonization was carried out in a tube furnace in a temperature range of 400^0 C to 600^0 C for half an hour. Carbonization was done in an inert atmosphere by providing nitrogen gas at a rate of 250 ml/min by using a nitrogen generator. Three different concentrations (10%, 20% and 30%) solution of phosphoric acid was prepared .A systematic investigation of the effect of concentration of acid and activation temperature, on the yield of activated carbon, was made. The surface area was measured using BET surface area analyzer. Iodine number was also calculated to find out the concentration of phosphoric acid and optimum temperature for carbonization. The FTIR analysis depicts the presence of variety of functional group. The porous surface of the activated carbon was observed by using Scanning electron microscope.

CHAPTER 1

INTRODUCTION

1. INTRODUCTION

Activated carbon is a special type of carbonaceous material that has been treated to create highly developed internal pore structure. Activated carbon, also widely known as activated charcoal. It is a form of carbon which has been processed to make it extremely porous and thus to have a very large surface area available for adsorption or chemical reactions.[1] Just 1 gm of activated carbon has a surface area in excess of 150 m^2. It is one of the best adsorbent known to us. High surface area of activated carbon is responsible for different useful applications; however adsorption properties can be enhanced by further chemical treatment.

Activated carbon, activated alumina, silica gel, metal organic frame work are some of the popular adsorbents used in industries. The most desirable property of adsorbent is large surface area (surface area per unit mass). The adsorptivity of adsorbent depends on the size of the molecule being adsorbed and also on the pore size of the adsorbent. These pores can be classified into three categories [2] :

1. Micro pores <2 nm
2. Meso pores 2-50 nm
3. Macro pores >50 nm

Any organic material having high carbon content can be used as a raw material for synthesis of activated carbon. There are two methods for further activation of these raw materials, physical and chemical. The temperature used in physical activation process is more than that used in chemical activation process. Chemical activation can be accomplished in single step by carrying out thermal decomposition of raw material with chemical reagent. Many cheap, easily available agricultural or plant based materials such as rice bran[3], straw[4], palm fiber[5], rubber wood saw dust[6], bamboo dust[7], date pits[8], palm fiber[9], coconut shells, groundnut shell, wheat husk[10] have been a source of production of activated carbon.

Physical activation is a process in which the raw material is activated by using gas or steam. Carbonization of the material containing carbon is pyrolized (heating in inert atmosphere) at high temperature (600 – 900^0 C) and then consequently activated by employing any oxidizing gas or steam. In another process, the raw material is directly exposed to activation at high temperature in presence of oxidizing gas in a single stage[11].

In chemical activation process, the raw material is impregnated with certain chemicals. Chemical activation processes have been carried out with acidic reagents, that is, $ZnCl_2$[12],H_3PO_4[13],HCl[14], and H_2SO_4[15] or with base reagents KOH[16], K_2CO_3[17], NaOH[18],

Na_2CO_3[19]. Among the various reagents, phosphoric acid has been widely used as it is less toxic, and has a good dehydrating property. After impregnation, the raw material needs to be carbonized at lower temperatures (450–900 °C). It is believed that the carbonization/activation step proceeds simultaneously with the chemical activation. Chemical activation is preferred over physical activation because of the lower temperatures and shorter time needed for activating material.

The wide application of activated carbon such as in the field of water purification, gas storage, separation etc. has resulted in increase in the demand of this material. Hence intensive research is needed in this field to explore the possibility of utilizing new precursor for the preparation of activated carbon. In this context, our research work is focused on studying the feasibility of the new precursor, an agricultural residue, Karanjia oil cake as an activated carbon. The work outlines the chemical activation method to prepare the activated carbon. Optimization of the process conditions such as concentration of the activating agent, impregnation ratio, activation temperature etc has been studied. Characterization of the surface properties of the developed activated carbon has been also examined.

1. 1 OBJECTIVE

The objective of this project work is

- To prepare a porous adsorbent from a low cost precursor.

Specific Objective is

- Preparation of activated carbon using phosphoric acid chemical activation process.
- Optimization of Process Parameter.
- Characterization of the Porous material.

CHAPTER 2

LITERATURE REVIEW

2. LITERATURE REVIEW

Activated carbon has been prepared from various cheap and easily available raw materials. And studies have been carried out by regarding synthesis and characterization of activated carbon from these raw materials. Various raw materials that have been used so far for production of activated carbon are enlisted below along with the authors.

Table1: List of raw materials which have been used for the production of activated carbon

Raw Material	Author
Bagasse and rice husk	DimitriosKalderis, Bioresource Technology[20]
Sugarcanebagasse	Girgis B.S.,Khalil L.B., TawfikT.A.M[21]
Corn Cob	W.T. Tsai, Bioresource Technology[22]
Rubberwoodsawdust	SrinivasakannanC.,BakarM.Z.A[23]
Rice Bran	R M Suzuki, J C Sousa, M C Rollemberg[24]
Bael Shell	J Anandkumar,BMandal[25]
Neem husk	Alau K. K, Archives of Applied Science Research[26]
bamboo	A.W.M. Ip, Bioresource Technology[27]
Coconutshell	LaineJ.,CalafatA.,LabadyM[28]

Bagasse and rice husk can be used for synthesis of activated carbon. The raw material is impregnated with phosphoric acid and then carbonized at a temperature range of 300-600^{0}C for maximum 3 hours. Activated carbon prepared from bagasse showed high surface area than that from rice husk. Maximum surface area for bagasse activated carbon was 1100 m^2/gm. and that of the other was 780 m^2/gm. Iodine number between 608 and 746 and methylene blue uptake of 213-261 (mg/g) were determined for the sample.

Activated carbon of iodine number of1096 mg/g and surface area of 1496 m^2/g and yield of 35% was obtained from rubber wood sawdust by following the standard procedure [23].In the 1st stage raw material was mixed with 60% phosphoric acid and soaked for overnight. Then semi carbonization was done by heating it in a muffle furnace at 200^{0}C for 15min. After 15 min the sample was taken out and again put in the furnace till it achieve desired temperature for carbonization (500^{0}C for 45 min).when carbonization was completed the product was washed first with distilled water and then with 0.1 M NaOH solution and dried in an oven at 105^{0}C.

Rice bran activated carbon has a BET surface area of 652 m^2/gm and a pore volume of 0.137 cm^3/gm. At first raw rice bran was mixed with water and some spherical particle and dried at a temperature of 100^{0}C for 5 hours. Spherical minimizes the loss of lighter particles. The dried sample was then carbonized under nitrogen gas flow at 200 mL min^{-1} and at a temperature of 850 °C, with a heating rate of 14 °C min^{-1}.The sample was heated at the carbonized temperature for 1 hour. The carbonized material was activated by a continuous flow of carbon dioxide at 850^{0}C for different period of times (40-120 min)[24].

For bael shell activated carbon, at first the raw sample was washed thoroughly to remove water soluble impurities and surface adhered particles. The it was dried in the hot sir oven at 110^{0}C .It was crushed by hammer mill and sieved in the size range of 600–860 μm. Chemical activation was done by using ortho phosphoric acid and dried at 110^{0}C for 2-3 hours. Calcination of the dried sample was carried out in the muffle furnace, starting from room temperature to a temperature of 600^{0}C. for 1.5 hours and then soaked in 2% $NaHCO_3$ to remove the residual acid. Finally, the adsorbent was dried at 110 °C for 2 h and cooled in a desiccator. In case of coconut shell activated carbon the raw material was brought to a size range of 0.2-0.5 mm. Phosphoric acid was used for chemical activation and was carbonized in a muffle furnace for 1 hour. After carbonization the product was cooled, washed and dried at 120^{0}C. The effect of carbonization temperature was studied and best results were obtained at 450^{0}C.

2.1 Classification of Activated carbon

Classification of activated carbon is made on the basis of their physical characteristics.

2.1.1 Powdered activated carbon (PAC)

PAC is fine granule whose size is less than 1 mm. Powdered activated carbon has high surface area to volume ratio. Its demerit is high pressure loss in applications. So, PAC is not used generally. They are prepared by finely grounded raw material and average pore size is between 0.15 to 0.25 mm.

2.1.2 Granular activated carbon (GAC)

Granular activated carbon is used for water treatment, deodorization and separation of components in a flow system. Rate of diffusion of GAC are faster, so they are preferred for all liquid and vapors adsorption. It is comparatively larger in size than powdered activated carbon. GAC exhibits a lower surface area than PAC.

2.1.3 Extruded activated carbon (EAC)

Extruded activated carbon is the result of fusion of powdered activated carbon by using a binder. EAC is cylindrical in shape and bigger in size. They are used for gas phase applications due to their high mechanical strength, low dust content and low pressure drop during applications. They have diameter ranging from 0.8 to 130 mm.

2.1.4 Impregnated Carbons

Inorganic metals like iron. zinc, lithium, calcium and manganese are impregnated upon carbon for specific applications. These carbons are used for air pollution control. Due to antimicrobial/antiseptic properties, silver loaded activated carbon is used as an adsorbent for purification of domestic water. Drinking water can be obtained from natural water by treating the natural water with a mixture of activated carbon and $Al(OH)_3$,a flocculating agent. When the carbon is impregnated with specific kind of antibacterial or antiseptic agent, it can be used in water purification processes.

2.1.5 Polymer coated carbon

This is a process by which a porous carbon can be coated with a bio compatible polymer to give a smooth and permeable coat without blocking the pores. It finds its application in hemoperfusion and other medical related adsorption operations. Hemoperfusion is a treatment technique in which large volumes of the patient's blood are passed over an adsorbent substance in order to remove toxic substances from the blood.

CHAPTER 3

MATERIALS AND METHODS

3. MATERIALS AND METHODS

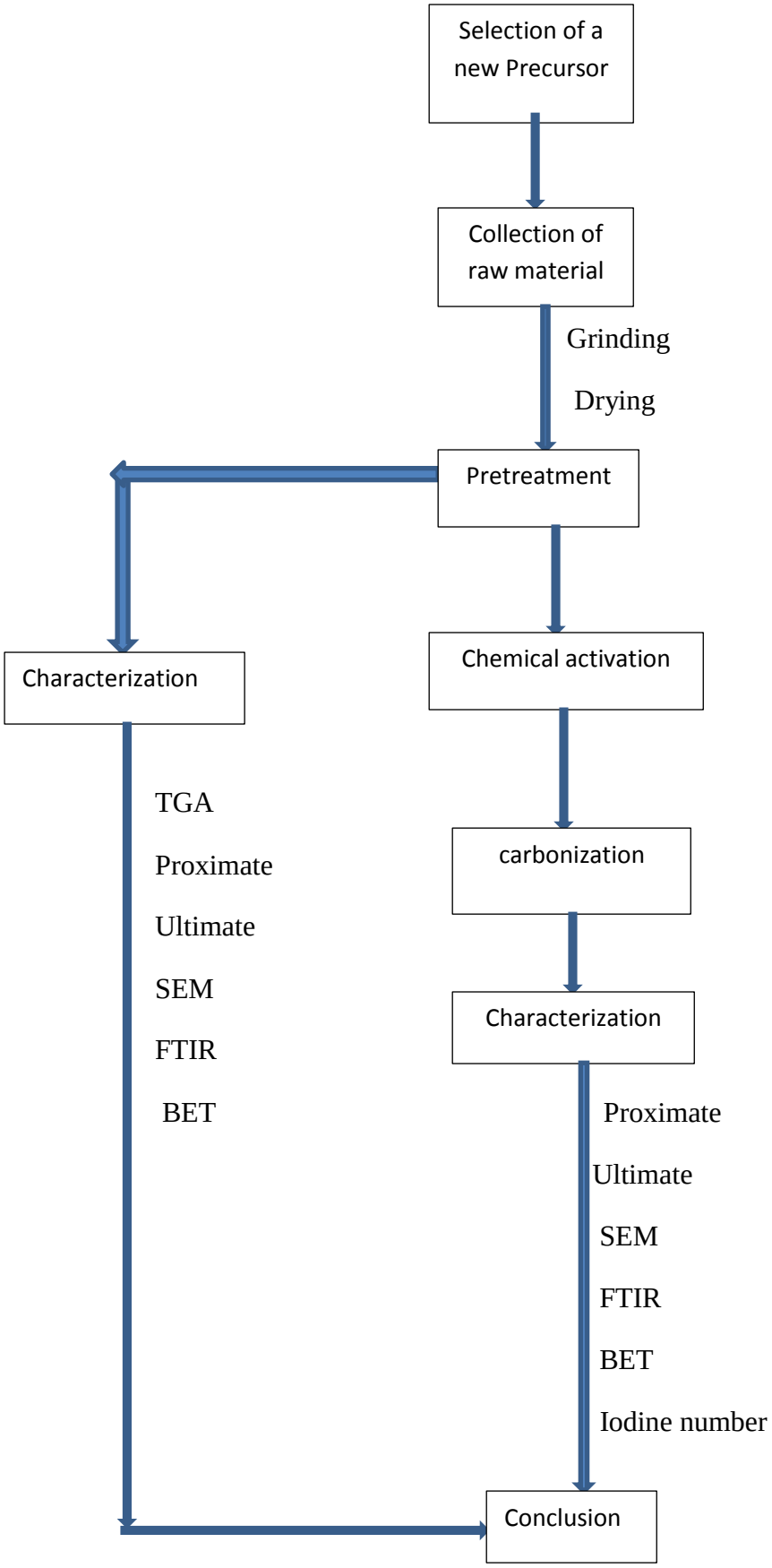

Fig.1-Methodologyoftheproject

3.1 Chemicals

A lot of chemicals have been used during the course of the project. They are ortho phosphoric acid for impregnation of raw material, iodine, sodium thiosulphate and starch for determination of iodine number, potassium bromide for FTIR analysis, liquid nitrogen cylinder for BET surface area analysis.

3.2 Instrumentation

The oil cake was grinded in an electrical grinder. Hot air oven was used for drying the impregnated sample and the activated carbon after washing. The carbonization of the sample was carried out in the Tube Furnace (OKAY 1400^0C electrical furnace, 40T 4Y). A shaker (Lab companion model SI-300R) was used in the process of determining iodine number. For ultimate analysis, CHNS analyzer (Elemental CHNS analyzer) has been used. Thermo Gravimetric Analyzer (TGA) (Schimadzu, DTG-60H) was used to determine the weight loss of the sample with rise in temperature. Scanning electron microscope (SEM-JEOL,JSM 6480LV) was used for visualization of surface texture of the precursor and the activated sample. BET apparatus (Autosorb, Blue star Ltd.) was used for the determination of specific surface area. Fourier Transform Infrared spectrometry (PerkinElmer, revolutionof4cm^{-1})was used to determine the organic functional groups present in the adsorbent.

3.3 Raw Material

Activated carbon sorption is highly effective in removal of dyes ,pigments as well as other organic and inorganic pollutions[].Conventionally, activated carbon is produced from carbonaceous source material such as wood, peat, coal, and wastes of vegetable origin (e.g. nutshells, fruit stones). This leads to searches for new, cheap and easily available resources of carbon as a feedstock for activated carbon. Karanja oil cake was the raw material used for synthesis of activated carbon. It was collected from a seed shop in ambagan market, Rourkela.

3.4 Pretreatment of Raw Material

The karanja oil cake was initially sundried to remove moisture. Then it was crushed by an electric grinder to very fine powder. The sample was then dried in an hot air oven to remove additional moisture. After complete drying, the raw material was ready for chemical activation. The precursor was initially characterized using proximate analysis, ultimate analysis, FTIR analysis, TGA, BET surface area analysis and SEM analysis.

3.5 Proximate Analysis

Proximate analysis is used for the determination of moisture content, volatile matter content, ash content and fixed carbon content of the sample by heating it under specific condition. It is defined by ASTM D 121. The proximate analysis of the precursor and activated carbon were done using the following procedure [29].

3.5.1 Moisture content

A known amount of the sample was taken in a petri dish and weighed using a weighing balance. The petri dish was placed in the hot air oven at 108^0C and dried for one and half hours. The sample was then taken out and cooled in a desiccator and weighed.

The moisture content M = 100(B-F)/ (B-G)

B= weight of petri dish + original sample

F= weight of petri dish + dried sample

G= weight of petri dish

3.5.2 Ash content

A known amount of sample was taken in a silica crucible. The crucible was ignited in the muffle furnace at 725^0C for one and half hour. Then it was taken out of the furnace, placed in the desiccator, cooled to room temperature and weighed.

The ash content A= 100(F-G)/ (B-G)

G= Mass of empty crucible

B=Mass of crucible + dried sample

F=Mass of crucible + ash sample

3.5.3 Volatile matter content:

A known amount of sample was taken in a cylindrical crucible. The crucible was placed in a muffle furnace at 925^0C, covered with lid, and heated for exactly 7 minutes. Then it was taken out, allowed to cool and weighed.

Volatile matter on dry basis VM=100[100(B-F)-M (B-G)]/ [(B-G) (100-M)]

B= Mass of crucible, lid and sample before heating

F= Mass of crucible, lid and contents after heating

G= Mass of empty crucible and lid

M= % of moisture as determined above

3.5.4 Fixed carbon content

The fixed carbon content is determined by subtracting sum of percentage compositions of moisture content, volatile matter content and ash content from 100.

Fixed carbon FC=100-(volatile matter + ash content)

3.6 Ultimate Analysis:

CHNS elemental analyzers provide a means for the rapid determination of carbon, hydrogen, nitrogen and sulphur in organic matrices and other types of materials. They are capable of handling a wide variety of sample types, including solids, liquids, volatile and viscous samples, in the fields of pharmaceuticals, polymers, chemicals, environment, food and energy.

This analysis determines the elemental composition of the sample and it is based o the principle of Duma's Method. This method involves complete and instantaneous oxidation of sample by flash combustion.

Sample is fed to the CHNS analyzer along with excess oxygen. Oxygen reacts with the elements present in the sample i.e. nitrogen, hydrogen, sulphur and carbon to produce nitrogen dioxide, water, sulphur dioxide and carbon dioxide. A chromatographic column is used for the separation of combustion products and these are detected by thermal conductivity detector (TCD). TCD gives an output signal proportional to the concentration of individual components. From this individual component of the elements in the sample is determined.

3.7 Fourier Transform Infrared Spectroscopy

FTIR is most powerful tool for identifying chemical bond (functional group).FTIR spectra of pure compounds are like a molecular fingerprint and organic compounds have very rich detailed spectra. But it is much simpler in case of inorganic compound. The spectrum of an unknown can be identified by comparison with a known compound.

A beam of infrared light is passed through the sample and the infrared spectrum is recorded. A part of this radiation is absorbed and other part is transmitted through the sample. Amount of energy that was absorbed by each wavelength can be found out by examining the transmitted light. Thus a spectrum is produced in the form of transmittance/absorbance plotted against the wavenumber. This spectrum detects the infrared wavelength at which absorption occurs. When the vibrational frequency of bond and frequency of IR are same, absorption occurs. Thus, it gives an idea about the organic functional groups present in the sample.

A pure KBr pellet and a pellet of sample mixed with KBr were prepared. KBr pellet was used as a reference. Then the sample pellet was placed in the device to obtain the IR spectrum in terms of transmittance v/s wavenumber plot.

3.8 Thermo Gravimetric Analysis

The decrease in weight of a sample with respect to time and temperature is determined by thermo gravimetric analysis. This serves as a basic study for activation process as it helps in determine working range of temperature for activation of sample. Maximum temperature, heating rate and holding time are the parameters required for analysis.

For the analysis, a small amount of sample was taken in the vial present in the analyzer and this vial is connected to a sensor which measures the weight of the sample throughout the analysis. Inert atmosphere was generated in the analyzer by using nitrogen. Heating rate was 10^0C /min and maximum temperature was set at 800^0C.

3.9 BET Surface Area Analysis

Brunauer, Emmett and Teller- Three scientist who proposed this theory and it is used to determine the surface area of porous powder. The principle lies in adsorption of gas molecules to the surface of solid whose surface area is to be measured.

BET theory is based on the following assumptions:

1. Gas molecules can be physically adsorbed on the solid surface and form infinite layers
2. There is no interaction between the layers
3. Langmuir theory is applied to each layer

The BET equation is given by:

$1/v[(P_0/P)-1]= (c-1)(P/P_0)/(v_mc)+1/(v_mc)$

P_0=Saturation pressure of the adsorbate

P=Equilibrium pressure of the adsorbate

v=Volume of gas adsorbed

v_m=Volume of gas adsorbed in the monolayer

c=BET constant given by exp $(E_1-E_L)/RT$

E_1=Heat of adsorption for the first layer

E_L=Heat of adsorption for higher layers

A plot of $1/v[(P_0/P)-1]$ v/s (P/P_0) is obtained from the BET analysis. From the slope and intercept of the line, v_m and c are obtained. Surface area S of the solid sample is given by

$$S= (v_mNs)/ (VX)$$

N= Avogadro's number

s=adsorption cross-section of the gas being adsorbed

V= molar volume of the gas being adsorbed

X= mass of adsorbent

About 0.1 gm of sample was taken in a tube and the tube was placed in dewar containing liquid nitrogen. At first the sample was purified by degasifying it. The surface area of the sample was determined based on adsorption of nitrogen gas.

3.10 Scanning Electron Microscope Analysis (SEM)

Scanning electron microscope uses highly energetic beam of light for produce the image of a sample by scanning it. Vacuum is maintained inside the microscope Electron beam strikes the sample placed in the sample chamber, gets decelerated and produces a variety of signals like secondary electrons, back scattered electrons, diffracted backscattered electrons, photons, visible light and heat. The detectors detect the secondary electrons and produce visible image of the surface of the sample.

Very small amount of sample was taken in a circular disc like structure and the sample was attached to the disc by a specific tape. Then sintering of the sample was done and the sample was coated with platinum. Then sample with the disc was placed in the electron chamber of the microscope. For better quality image and for proper view of the surface the magnification of scan was varied.

3.11 Chemical Activation

Chemical activation of the dried sample was done by using Phosphoric acid. Different concentration (10%, 20%,30%) of phosphoric acid solution was prepared. These solutions were added to the precursor so that all the particles were completely wetted. The samples were allowed toremainsoakedintheacidfor24hours after which the beaker was put in the hot air oven till the acid was completely dehydrated to give dried black granular structures. The time required for drying depends on the concentration of phosphoric acid and amount of sample taken.

3.12 Carbonization

Tube furnace was used for carbonization of impregnated sample. Inert atmosphere was provided by supplying nitrogen gas continuously by using a nitrogen generator. Nitrogen flow rate was maintained at 250 ml/min. In the tube furnace heating rate was set at 4^0C/min. The sample was kept in the middle of the tube of the tube furnace (shown in figure). The activation temperature and time was changed as and when required. The samples were then taken out, washed with distilled water. Then it was dried in the hot air oven at 60^0C till it was completely dried.

Fig.2-Tube Furnace used for carbonization

3.13 Iodine Number

Iodine number is a measure of the micro pore content of the activated carbon. More is the iodine number; more is the micro porosity of the sample. It is the milligram of iodine adsorbed by 1 g of activated carbon from 0.1 N iodine solutions. Iodine number was determined by following procedure [30].

About 0.2 g of powdered activated carbon was taken and 40 ml of 0.1 N iodine solutions was introduced to it. Then the content was put in a shaker for exactly 4 min. After shaking the content was filtered through whatman filter paper and 10 ml of the filtrate was titrated against standard sodium thiosulphate solution.0.05 N sodium thiosulphate solution was taken. Indicator used was startch solution. After titration concentration of resulting solution was calculated and from this iodine number was determined.

CHAPTER 4

RESULTS AND DISCUSSION

4. Results and Discussions

4.1 Thermo Gravimetric Analysis

The TGA result of the precursor indicates that initial weight loss starts at 200^0C, but the drastic change in weight occurs between 350^0C to 600^0C .Maximum removal of volatile matter and moisture were taken place in this temperature range. The constant line towards the end of the plot represents the constant ash that is left over.

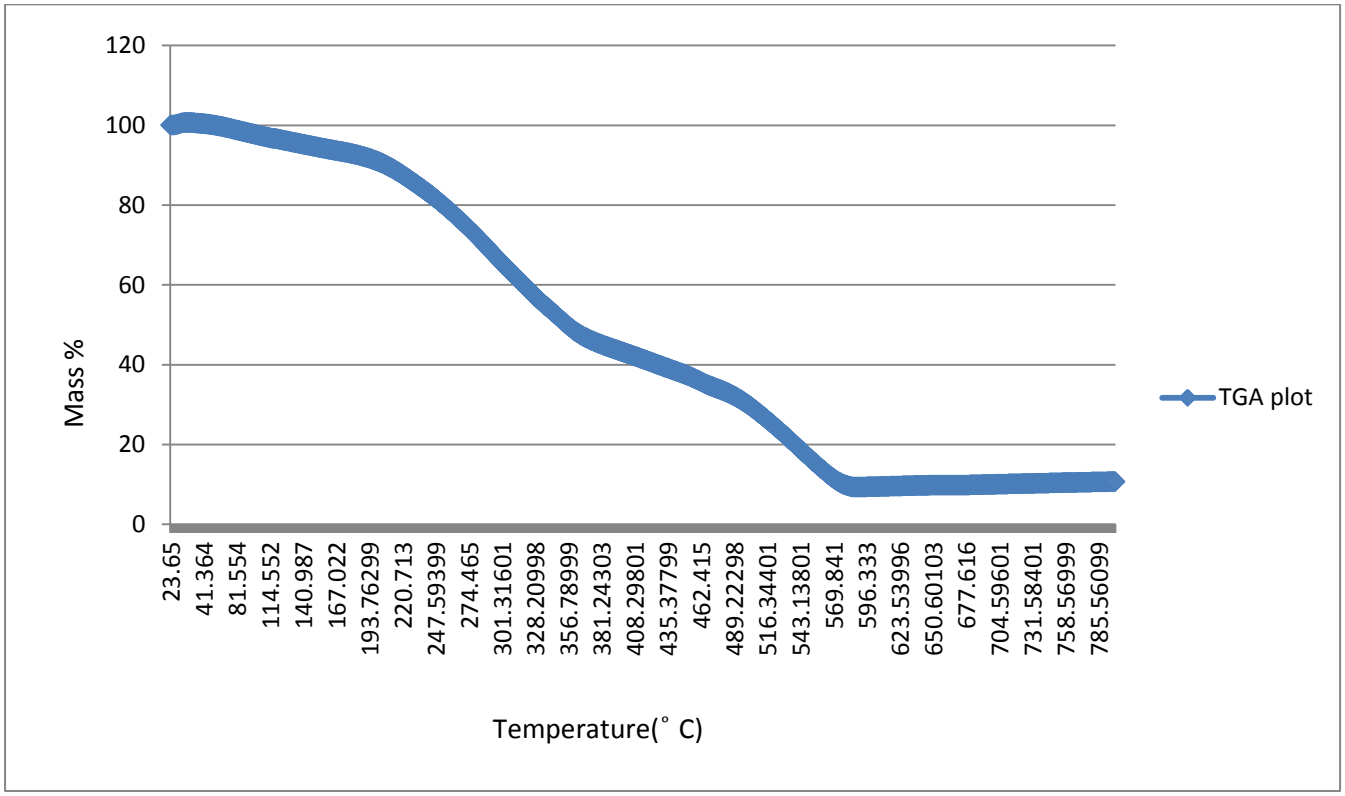

Fig-3 TGA thermograph of the chemically treated precursor

4.2 Proximate Analysis

Result of proximate analysis of the precursor and the activated carbon are shown in the following table no. 2.

Table 2 – Proximate analysis of precursor and activated carbon

Parameters	Raw sample	Activated carbon
Moisture (%)	2.25	1.36
Volatile matter (%)	79.32	18.78
Fixed carbon (%)	14.7	76.02
Ash (%)	3.73	3.84

From the above table, the precursor has a high volatile matter content and lower carbon content. After carbonization there is substantial increase in carbon content and decrease in volatile matter. Most part of the volatile matter was lost due to heating of the sample and increase in moisture content may be due to washing involved in the process of carbonization. The increase in ash content results from the increase in the inorganic content of the sample which may be due to addition of phosphoric acid.

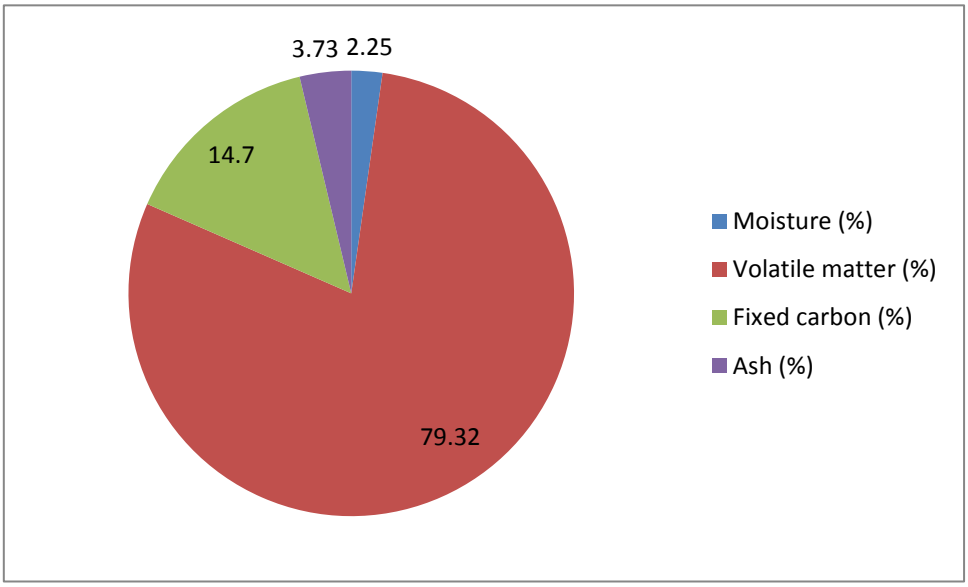

Fig 4-Proximate analysis of precursor

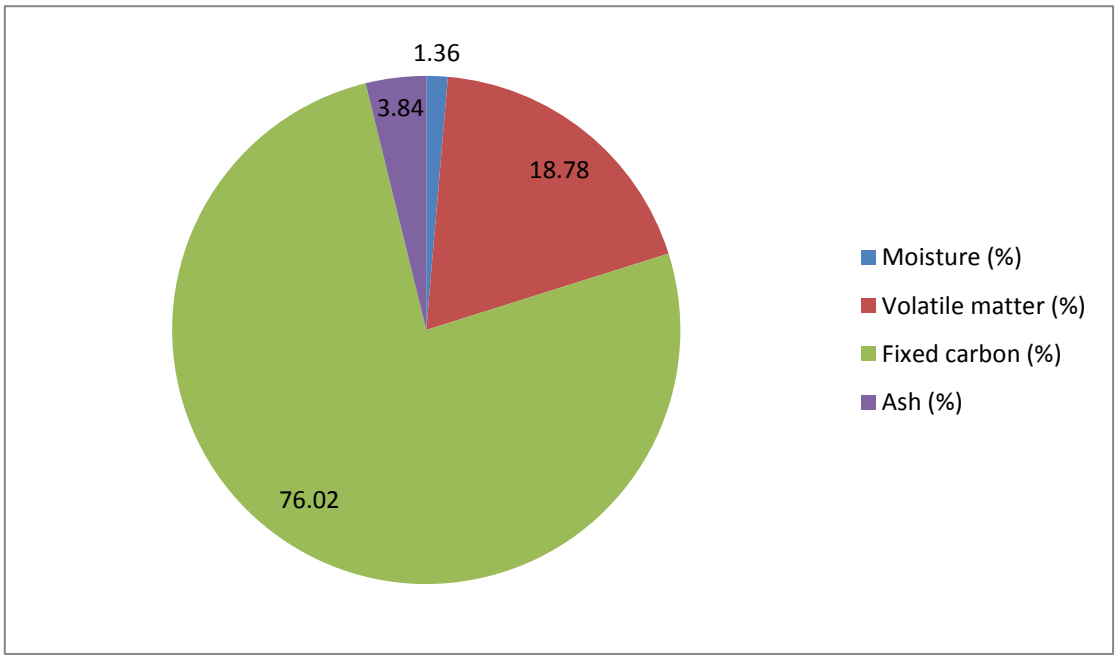

Fig 5- Proximate analysis of activated carbon

4.3 SEM Analysis

SEM images are used to determine the structure and distribution of pores on the surface of the activated carbon and precursor. The SEM images of precursor and activated carbon produced from it are shown below.

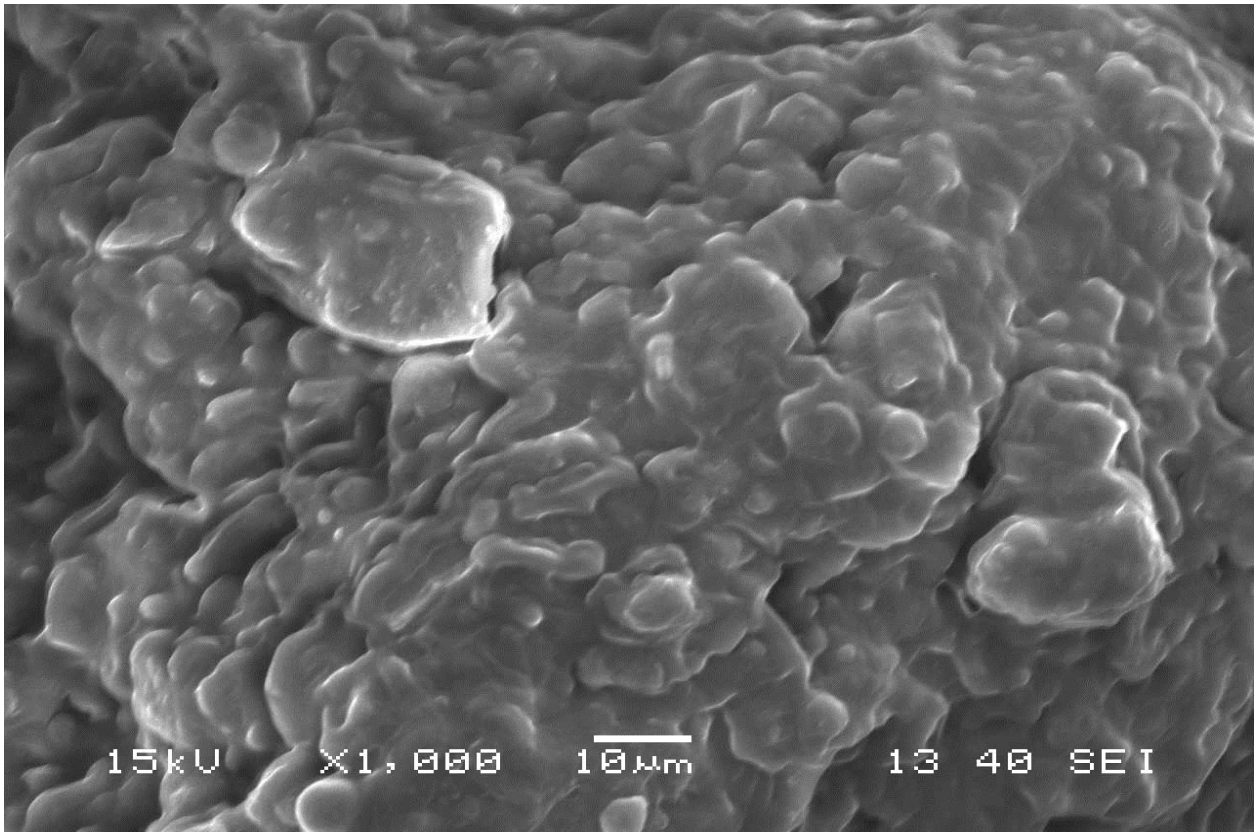

Fig 6 – SEM image of the precursor

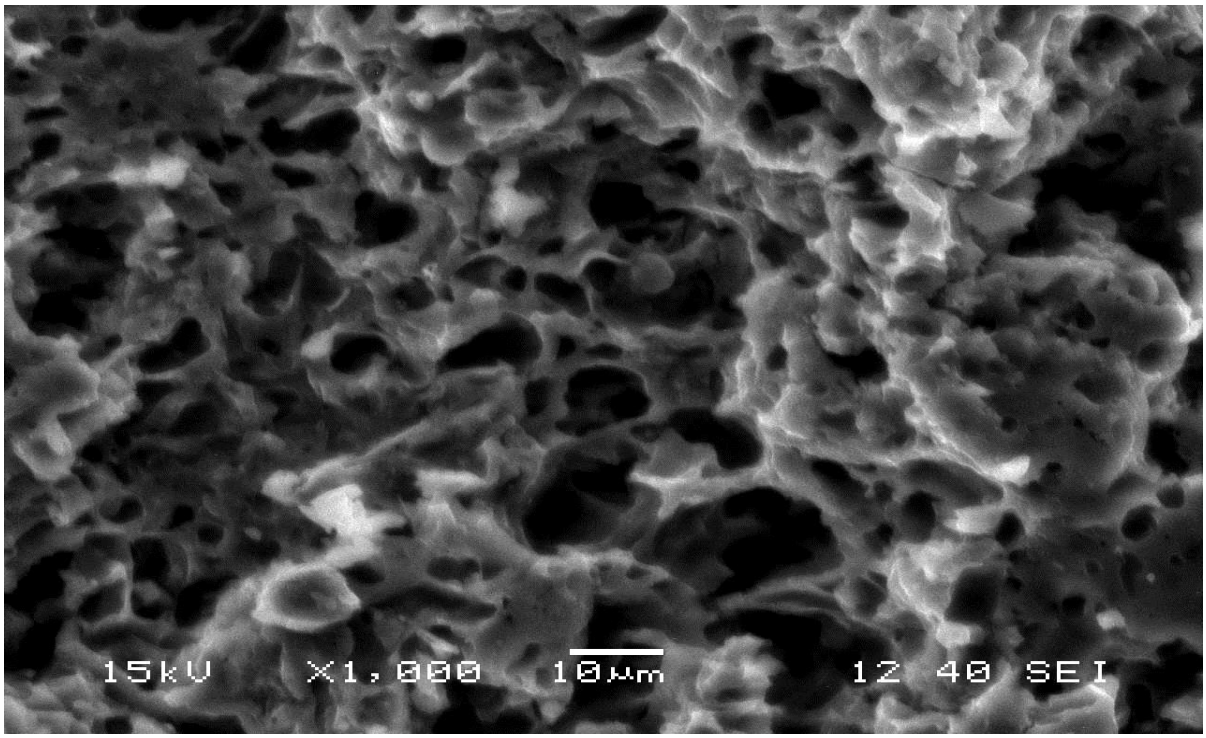

Fig 7 – SEM image of the activated carbon

As seen from the above two figures more number of pores are visible in the activated carbon, but in case of the precursor there are no pores visible at all. This supports the fact that the activated carbon has a highly developed porous structure.

4.4 Iodine Number

Table 3 – Iodine number of the activated carbon prepared at different temperature

Sample		Concentration of iodine after adsorption(N)	Quantity of iodine after adsorption(Q) Q=(127*N*40)/1000	Quantity of Iodine adsorbed(q)(gm) q=0.523-Q	Iodine adsorption q/0.2 (mg/gm)
Temp(^{0}C)	% impregnation				
400	10	0.004	0.02032	0.5	2500
	20	0.0035	0.01778	0.5052	2526.1
	30	0.0035	0.01778	0.5052	2526.1
450	10	0.0025	0.0127	0.5103	2551.5
	20	0.002	0.01016	0.52184	2564.2
	30	0.002	0.01016	0.52184	2564.2
500	10	0.0025	0.0127	0.5103	2551.5
	20	0.0015	0.00762	0.51538	2576.9
	30	0.001	0.00508	0.51792	2589.6
550	10	0.002	0.01016	0.52184	2564.2
	20	0.002	0.01016	0.52184	2564.2
	30	0.0015	0.00762	0.51538	2576.9
600	10	0.0045	0.02286	0.50014	2500.7
	20	0.0035	0.01778	0.5052	2526.1
	30	0.0025	0.0127	0.5103	2551.5

Iodine number gives an idea about the micro porosity of activated carbon. Here the iodine number was calculated for all the 15 samples which are shown above in the table. Variation of iodine number with temperature for different concentration of phosphoric acid can be observed by plotting graph between them.

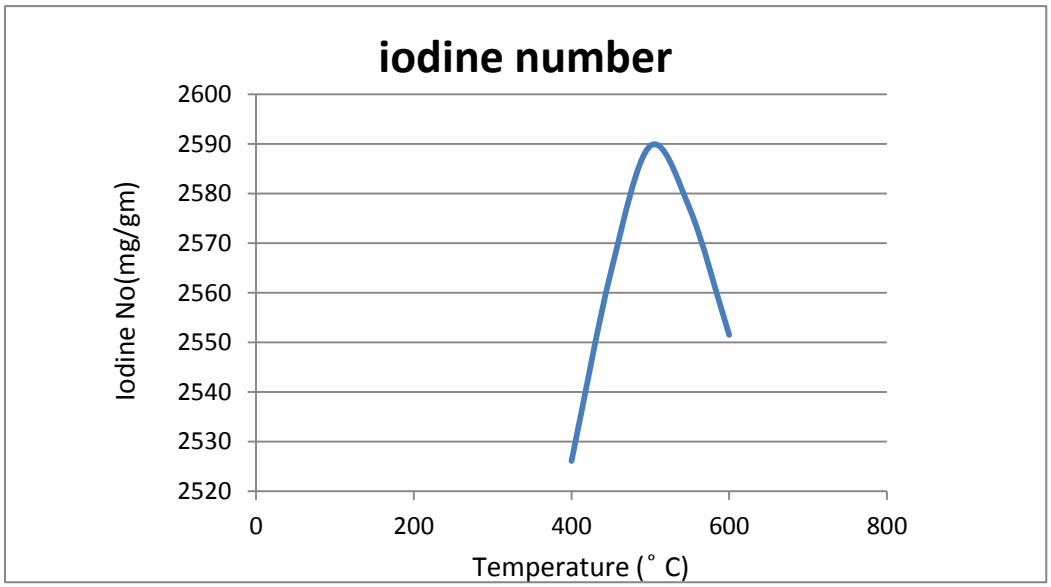

Fig 8 – Variation of iodine number with temperature for 10% concentration of acid

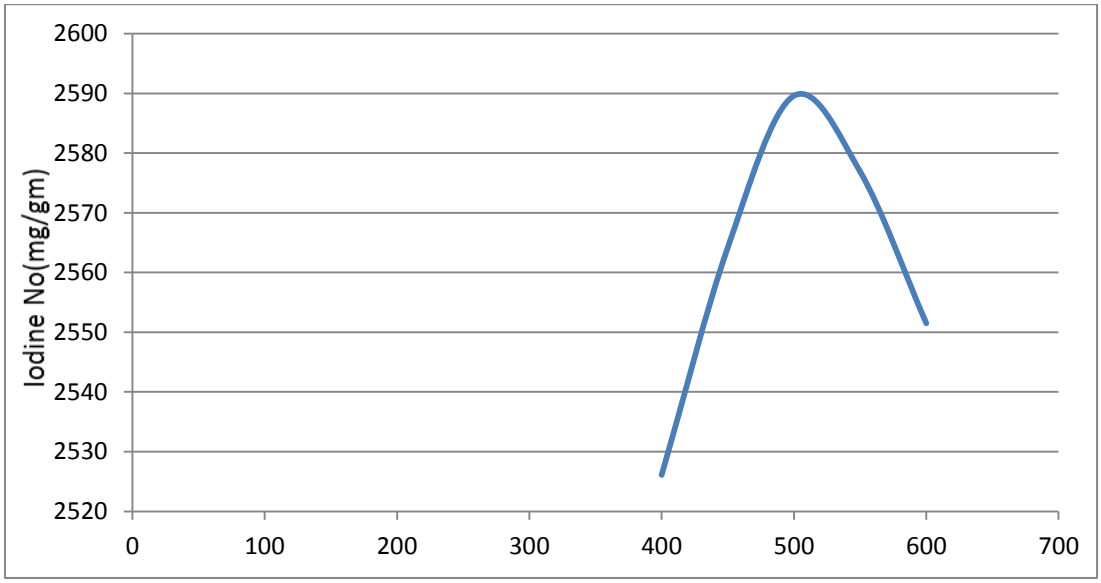

Fig 9 – Variation of iodine number with temperature for 20% concentration of acid

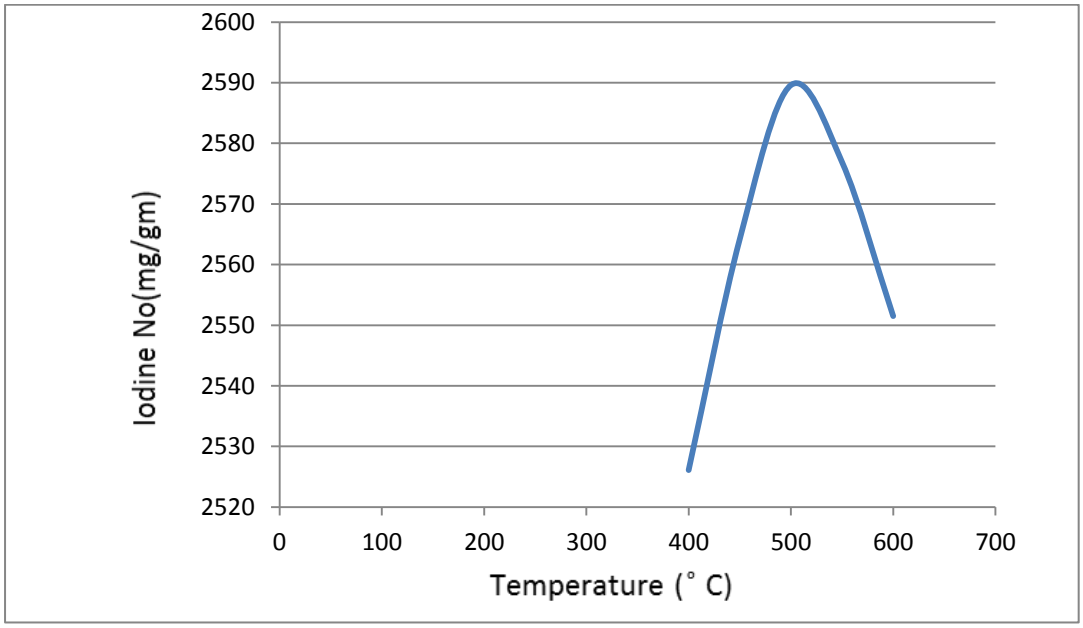

Fig 10 – Variation of iodine number with temperature for 30% concentration of acid

Maximum value of iodine number was determined as 2589.6 mg/gm of activated carbon at 500^0C for 30% concentration of phosphoric acid. Iodine number increases with increasing in temperature till a certain temperature. This may be due to evolution of volatile matter on increasing temperature, but at higher temperature there is a possibility of cracking of material .So, there is no further adsorption of iodine at higher temperature.

4.5 Yield

Yield of the activated carbon is determined by varying the temperature and time required for carbonization. Following table consists of yield at different temperatures and at different concentration of phosphoric acid.

Table 4 – Yield of activated carbon at different temperature and concentration of acid

Temperature (in ^{o}C)	% yield of the impregnated sample in 10% concentration of phosphoric acid	% yield of the impregnated sample 20% concentration of phosphoric acid	% yield of the impregnated sample 30% concentration of phosphoric acid
400	74	71.3	69.2
450	73.2	69.5	64.6
500	68.4	63.2	59.1
550	62.7	57.7	52.9
600	55	53	52

Yield decreases on increasing concentration of phosphoric acid. Phosphoric acid tends to produce dehydrating effect on cellulose, hemicellulose and lignin in the interior of the particles during heat treatment [31].The acid tends to break the ligno-cellulosic bonds of the precursors and release the non-carbon elements in terms of liquids (tars) and gases leaving behind a rigid carbon skeleton [32]. More liquid and gaseous products formed on increasing concentration of acid which leads to lower yield. Again on increasing temperature more removal of volatilities occur which also leads to lower yield.

Effect of temperature on the yield can be observed from the following plot:

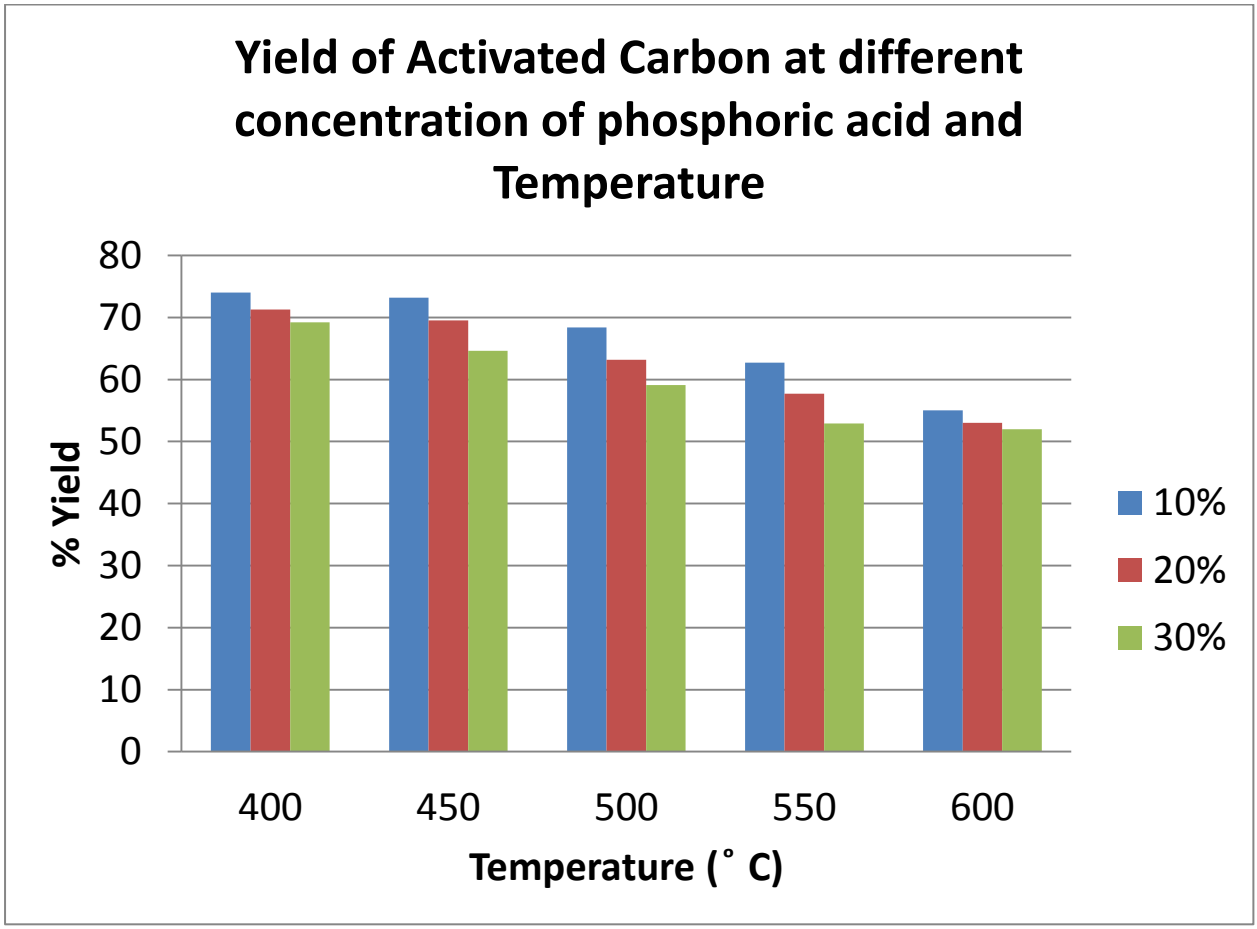

Fig 11 – Yield of activated carbon at different concentration of phosphoric acid and temperature

4.6 BET analysis

Higher surface area represents better adsorption capacity. BET surface area analysis is a method of determining the adsorptivity of the sample. From the surface area analysis, the precursor has a surface area of 49 m^2/gm whereas activated carbon has a surface area up to 179 m^2/gm. The effect of acid concentration on the BET surface area was studied. The following table shows the surface area corresponding to phosphoric acid concentration.

Table 5- BET surface area corresponds to different concentration of acid

% concentration of phosphoric acid	BET surface area (m^2/gm)
10	118
20	139
30	179

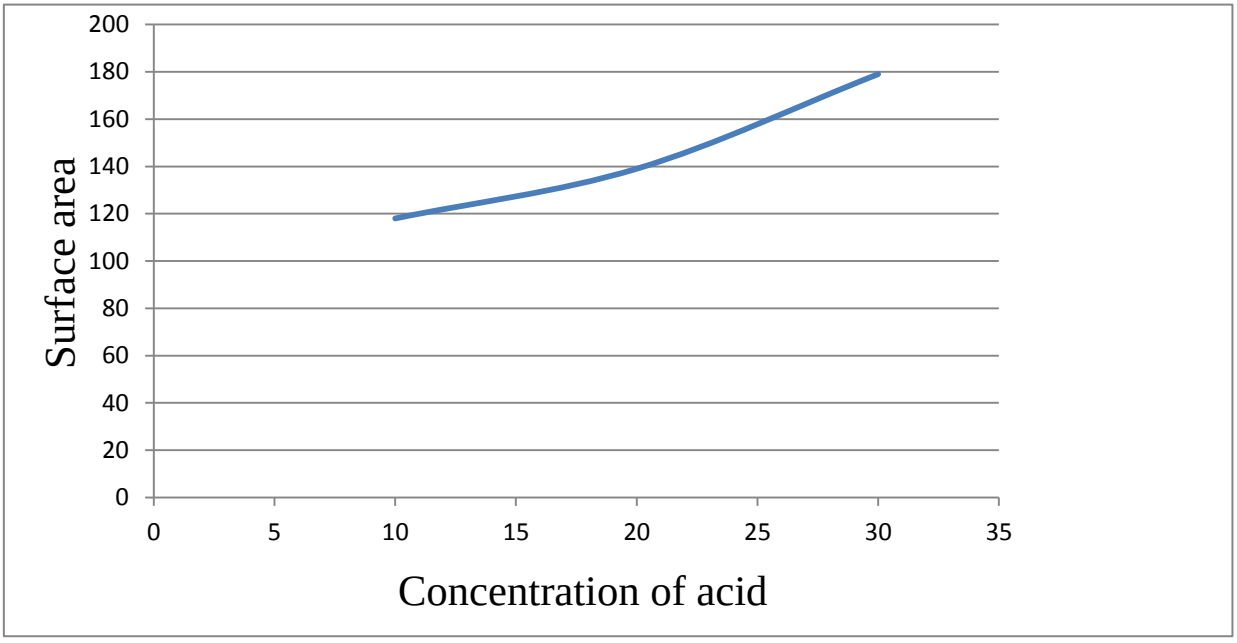

Fig 12 - Concentration of phosphoric acid vs BET surface area

4.7 FTIR Analysis

Fourier transform infrared spectroscopy (FTIR) is used for identification of organic and inorganic compounds' functional groups present in the compound, components of the unknown mixture. It is called Fourier transform infrared spectroscopy because Fourier transform, a mathematical process is required to transfer the raw data into actual spectrum.

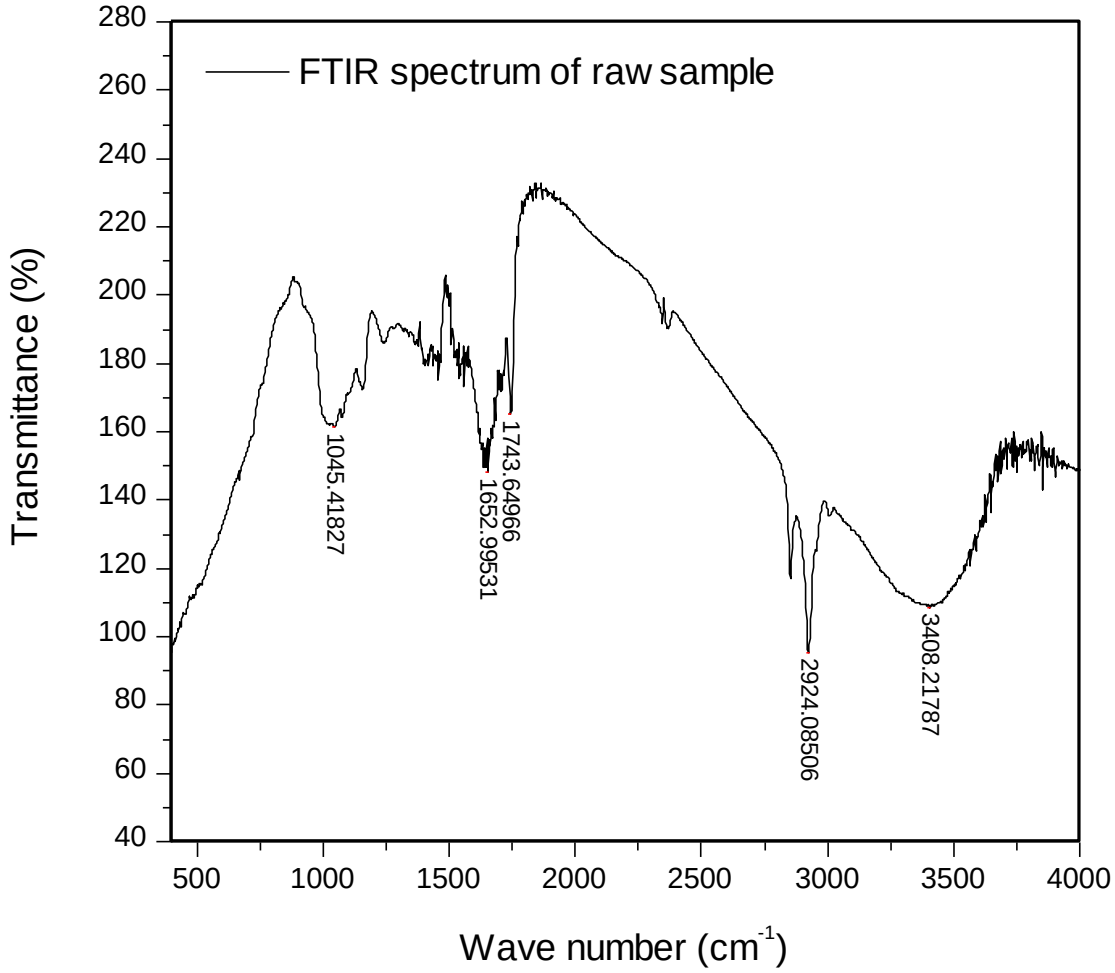

Fig 13 – FTIR Spectra of raw sample

Some peaks were found in the characterization of raw data sample. 1045 cm^{-1} corresponds to C-O bond of ether, 1653 cm^{-1} wave number revealed the C=C stretching vibration, 1744 cm^{-1} corresponds to carbonyl stretching frequency, 2924 cm^{-1} has the significance of SP^3 C-H bond vibration, 3408 cm^{-1} corresponds to O-H stretching bond vibration of water.

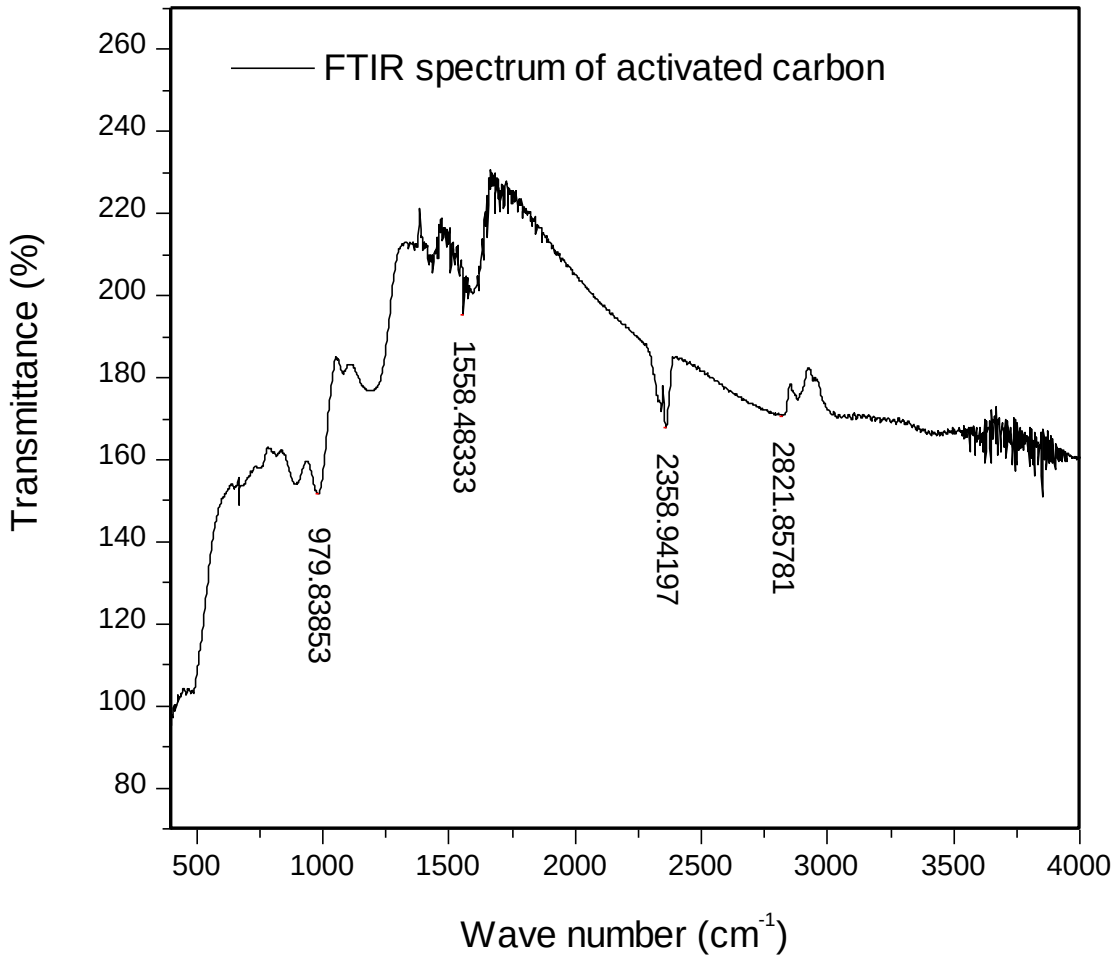

Fig 14- FTIR spectra of activated carbon

The peaks found from the characterization of prepared activated carbon have various significances. 980 cm^{-1} corresponds to stretching vibration of C=C cis alkene. 1558 cm^{-1} assigns to C=N bond vibration, peak at 2359 cm^{-1} is for C-H bond stretching vibration, 2822 cm^{-1} indicates the presence of C-H bond of alkane.

CHAPTER 5

CONCLUSIONS

5. CONCLUSION

Proximate analysis of the precursor gives an idea about the physical properties of the sample. It helps in choosing the appropriate precursor for synthesis of activated carbon. According to proximate analysis the raw material has a carbon content of about 14 % and combined moisture and volatile matter content of about 81 %. So there are more amount of easily escapable components present in karanja oil cake. Proximate analysis also shows that the fixed carbon content of the activated carbon was high.

Temperature range of activation was determined by thermo gravimetric analysis. With the help of this result proper carbonization time and optimum temperature was determined.

The variation of BET surface area with phosphoric concentration was studied. Three different concentration of phosphoric acid (10%,20%,30%) were used for impregnation of the raw sample. It was found that surface area increases on increasing concentration of phosphoric acid. Maximum surface area of 179 m^2/gm was obtained at the phosphoric acid concentration of 30%.

For identifying the optimum temperature iodine number of various activated carbons were determined. Iodine number is the measure of micro porosity of the activated sample. Maximum iodine number of 2589.6 mg/gm. of activated carbon was obtained at a temperature of 500^0C with 30% phosphoric acid impregnation.

The effect of temperature and concentration of activated carbon on the yield of the activated carbon was determined. It was found that yield decreases with increase in both temperature and concentration of acid.

Scanning electron microscope images gives a view about the pores present on the surface of activated carbon. From figure 6 and 7 it is inferred that, there was hardly a pore visible on the surface of raw material, but there were clearly visible pores in case of activated carbon.

CHAPTER 6

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REFERENCES

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